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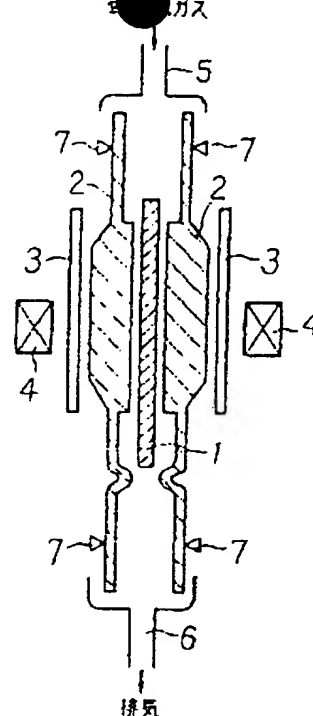
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TITLE : PRODUCTION OF OPTICAL FIBER
PREFORM



ABSTRACT : PURPOSE: To produce the title large-sized preform free of deformation by fusing a core and a clad to each other with an electric furnace, and specifying the operational conditions in the production of the preform by the rod-in-tube method.

CONSTITUTION: A core glass rod 1 having a diameter smaller than the inner diameter of a clad glass pipe 2 having ≥ 50 mm outer diameter is inserted into the glass pipe 2. The obtained material is heated at $1800\sim 2000^{\circ}\text{C}$ in an electric furnace (furnace core tube 3) having a heater 4 $30\sim 100$ mm in length while traversing the furnace at a velocity of $5\sim 20$ mm/min. The glass pipe 2 and the glass rod 1 are thereby fused together, and a glass rod is obtained. During heating, the hollow part of the glass pipe 2 is preferably evacuated to $50\sim 300\text{mmHg}$ below atmospheric pressure.

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(54) Subject of Invention Manufacturing Method of Preform for Optical Fiber

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DETAILED DESCRIPTION

1. Subject of Invention

Manufacturing method of preform for optical fiber

2. Scope of the Patent Claim

- (1) A manufacturing method of preform for optical fiber having the following characteristics: Into the hollow portion of a glass pipe for the clad of more than 50 mm in outside diameter, a glass rod for the core of diameter smaller than the inside diameter of the pipe for the clad is inserted; while this (assembly) is traversed at speed of more than 5mm/min but less than 20 mm/min in an electric heater of more than 30 mm but less than 100 mm in length, it is heated at heater temperature 1800—2000°C; by this the glass pipe for the clad and the glass rod for the core would become one body to yield a glass rod possessing the core and the clad.
- (2) In the manufacturing method of preform for optical fiber described in Claim Item (1), Scope of the Patent Claim, the heating is performed under the condition that the atmospheric pressure inside the hollow portion of the glass pipe for the clad is reduced by 50—300 mm H₂O (atmospheric pressure difference).
- (3) In the manufacturing method of preform for optical fiber described in Claim Item (1) or (2), Scope of the Patent Claim, the heating is performed under the condition that the atmosphere inside the hollow portion of the glass pipe for the clad is set to be an atmosphere containing chlorine or chlorine compound gas.
- (4) In the manufacturing method of preform for optical fiber described in Claim Item (1), Scope of the Patent Claim, the glass rod for the core is composed of pure quartz and the glass pipe for the clad is composed of fluorine doped quartz glass whose

refractive index is lower than that of pure quartz by more than 0.25% in specific refractive index difference.

3. Detailed Explanation of the Invention

[Industrial Application Field]

The present invention is related to a manufacturing method of preform for large type optical fiber.

[Conventional Technology]

In the manufacturing methods of preform for optical fiber, the rod in tube method in that into the pipe to become the clad material, a glass rod for the core possessing a refractive index higher than that of the clad material is inserted; the assembly is heated for consolidation (collapsing) to manufacture a preform for optical fiber is known as a representative manufacturing method.

However, in this method, defects (blisters, impurities, etc.) at the interface between the core material and the clad material can easily remain. Thus there is a shortcoming that when it is made into optical fiber, large light loss would occur. As a method for solving this, in Patent Public Notice Bulletin No. 59-6262 (1984) and Patent Public Notice Bulletin No. 58-52935 (1983), methods have been proposed that prior to the fusion-collapsing of the core material and the clad material, a vapor phase treatment agent is flowed in the space between the pipe and the rod and a prior heat treatment at the temperatures range of 500—1600°C where no deformation would occur to the core material is performed.

[The Problematic Point to be Solved by the Invention]

Now, in the conventional rod in tube method, as a heating means for consolidating the core material and the clad material, an oxyhydrogen flame has been employed. However, the heating by oxyhydrogen flame has a limit in its efficiency; at most, only preforms of about 30 mm in outside diameter can be obtained.

In recent years, reduction in cost by mass production in the manufacturing of optical fiber is being pursued; preparation of large type preform is being examined. The limitation to the preform outside diameter as described above is one of the bottle necks; thus its rapid solution is being demanded.

Against this, the electric furnace for example by using resistance furnace, etc. which is higher in heating efficiency than that of the oxyhydrogen flame and would also eliminate the worry for hydroxyl group occlusion can be considered. However, when a resistance furnace is used, the deformation of the preform would be more pronounced; thus this approach has not achieved practical application.

The present invention is undertaken in view of the above. For the heating means in the rod in tube method, an electric furnace is used in that a large type preform can be manufactured without deformation to provide a method which is very practical, economical and enhanced in process efficiency.

[The Means and Function for Solving the Problematic Point]

The present inventors carried out penetrating studies on the conditions in that even if an electric furnace is used as the heating means to carry out the consolidation (collapsing) of the core and clad by the rod in tube method, the preform can be manufactured without deformation. It was discovered that the aforementioned objective

can be achieved for the first time by specific combinations of the core material, clad material, and each of their sizes and the heating method and condition

Namely, the present invention is a manufacturing method of preform for optical fiber having the following characteristics: Into the hollow portion of a glass pipe for the clad of more than 50 mm in outside diameter, a glass rod for the core of diameter smaller than the inside diameter of the pipe for the clad is inserted; while this (assembly) is traversed at speed of more than 5 mm/min but less than 20 mm/min in an electric heater of more than 30 mm but less than 100 mm in length, it is heated at heater temperature 1800—2000°C; by this the glass pipe for the clad and the glass rod for the core would become one body to yield a glass rod possessing the core and the clad. In the aforementioned method, as an especially preferable implementation mode, the heating is performed under the condition that the atmospheric pressure inside the hollow portion of the glass pipe for the clad is reduced by 50—300 mm H₂O (atmospheric pressure difference). And, in the aforementioned method, the heating is performed under the condition that the atmosphere inside the hollow portion of the glass pipe for the clad is set to be an atmosphere containing chlorine or chlorine compound gas. And in the present invention, an especially preferable combination of the core and rod is that the glass rod for the core is composed of pure quartz and the glass pipe for the clad is composed of fluorine doped quartz glass whose refractive index is lower than that of pure quartz by more than 0.25% in specific refractive index difference.

The present invention is concretely illustrated by referring to figures below. Fig 1 is an outline of illustration diagram showing an implementation mode of the present invention. In the figure, 1 is the glass rod for the core; 2 is the glass pipe for the clad; 3 is

the furnace core tube; 4 is an electric furnace, for instance the heater of a resistance furnace, etc.; 5 is the atmospheric gas introduction opening; 6 is an exhaust opening; 7's are preform holders and the chucks for (performing) traversing. As shown in the figure, into the hollow portion of the glass pipe 2 for the clad, the glass rod 1 for the core is arranged; while this (the assembly) is held by the chucks 7 and being traversed, the two (the assembly) are heated by the heaters 4 of the electric furnace to become one body. During this, from the atmospheric gas introduction opening 5, a suitable atmospheric gas is introduced in carrying out the operation.

In the present invention, the glass pipe for the clad is composed of a glass having refractive index lower than that of the glass rod for the core; and it is especially preferable that the outside diameter is more than 50 mm. When the outside diameter is less than 50 mm, by the scattering of impurities from the heater 4 of the electric furnace, etc. if the outside wall of the glass pipe for the clad is contaminated, the effect of this pipe outside wall would easily appear. And the glass rod for the core is composed of a glass possessing refractive index higher than that of the glass pipe for the clad and the diameter (outside diameter) is smaller than the inside diameter of the glass pipe for the clad. Preferable combination for these glass rod for the core and the glass pipe for the clad includes, for example: pure quartz for the core glass rod and fluorine doped quartz glass for the clad glass pipe. The reason for this is that fluorine doped quartz glass is smaller in refractive index than that of the pure quartz glass and it does little to cause degradation to the property of the light transmission loss. Therefore, it can be used as an extremely superior clad material against the quartz glass core. And, when this combination is used to prepare a single mode fiber, it is preferable that the fluorine doped quartz glass is set to

be lower in refractive index than that of pure quartz by more than 0.25% in specific refractive index difference. By this, a fiber which is strong against bending loss and stable in transmission loss can be obtained. Furthermore, this kind of glass rod for the core and the glass pipe for the clad can be manufactured by the VAD method, OVD method, MCVD method, sol-gel method, press method, and other publicly known techniques.

In the present invention, the electric furnace to be used as heating source, for instance the electric resistance furnace, etc. is preferably; and the heater length has to be less than 100 mm and more than 30 mm. The heater is heated to a temperature of 1800—2000°C and then the assembled pipe and the rod are traversed at a speed of more than 5 mm/min and less than 20 mm/min. If the heater length exceeds 100 mm, preform deformation would occur; and when it is less than 30 mm, even if the outside wall of the glass pipe for the clad is raised to high temperature, it would be difficult to sufficiently raise the temperature of the inside wall of the glass pipe for the clad or the surface of the rod for the core. If the temperature is less than 1800°C, similarly, the surface temperature of the inside wall of the glass pipe for the clad or the surface temperature of the rod for the core would not be raised sufficiently; this is not desirable. And if the temperature exceeds 2000°C, the deformation of the glass pipe for the clad would abruptly become larger. When the traverse speed is less than 5 mm/min, deformation would occur to the preform and when it exceeds 20 mm/min, blisters would be formed in the preform. As a result, the performance of the optical fiber obtained from this preform would be degraded—this is not desirable.

During the heating in the present invention, it is preferable that it is performed under the condition that the atmospheric pressure inside the hollow portion of the glass

pipe for the clad is reduced by 50—300 mm H₂O (atmospheric pressure difference). Especially, it is desirably to be reduced pressure of 100—300 H₂O. When it is less than 50 mm H₂O, sufficient consolidation (collapsing of the space) would be difficult to carry out; and when it exceeds 300 mm H₂O, the performance of the fiber would be degraded.

And during the heating it is especially preferable that the heating is performed under the condition that the atmosphere inside the hollow portion of the glass pipe for the clad is set to be an atmosphere containing chlorine or chlorine compound gas. By this, the dehydration of the glass rod for the core and the glass pipe for the clad would be sufficiently performed to obtain a glass rod in which the residual hydroxyl group content is reduced in highest degree can be obtained. In this, for the chlorine or the chlorine compound gas, for example, Cl₂, CCl₄, SOCl₂, etc. can be employed.

Fig 2 shows the equipment construction in that the chlorine or chlorine compound gas atmosphere and pressure reduction are provided: in addition to the Fig 1 construction, the exhaust opening portion 6 and the pressure reduction means 8 are provided. And, in between the exhaust opening 6 and the pressure reduction means 8, the pressure gauge 9 and the gas introduction valve 10 for adjusting the pressure are arranged. While chlorine or chlorine compound gas is being introduced into the hollow portion of the glass pipe for the clad from the atmospheric gas introduction opening 5, the pressure is reduced by the pressure reduction means 8. However, by the valve 10, the flow rate of the gas for the pressure adjustment is controlled to set the pressure of the pressure gauge 9 to the specified pressure. For this pressure adjustment gas, for example, N₂, Ar, He, etc. inert gas, chlorine, or SOCl₂, CCl₄, etc. chlorine compound gases can be employed.

In carrying out the above described operation, the present invention can manufacture optical preforms of high quality which are larger in diameter than those produced so far by using an electric furnace without deformation. The concrete means and the effect of the present invention are shown by the implementation examples and the comparison example listed below.

[Implementation Example]

Implementation Example 1

For the glass rod for the core, a pure quartz glass rod (5 mm diameter x 500 mm length) prepared by the VAD method (vapor axial deposition method) was used; and for the glass pipe for the clad, a quartz glass pipe (outside diameter 75 mm x inside diameter 10 mm x length 400 mm) doped with 1.1 wt% of fluorine (F) was used. Based on the present invention, the heating consolidation to one body was performed by the construction shown in Fig 2. In this, a heater in 60 mm length was employed. The heater temperature was set to 1970°C, as the atmospheric gas, 1 liter/min of Cl_2 was flowed into the pipe hole and the pressure inside the glass pipe hole for the clad was reduced so that the atmospheric pressure difference was maintained to be 250 mm H_2O . Under these conditions, the preform was moved upward at 8 mm/min speed to carry out heating consolidation into one body. After the completion of the ascending, the obtained preform was taken out from the furnace and elongated. Then by using a preform analyzer, the core diameter and the clad diameter were measured. Slight fluctuation in length direction was present; however, the fluctuation of the clad diameter/core diameter was less than 1%. An excellent preform without blisters in the core-clad interface was obtained. The refractive index difference of the core and clad was 0.30%.

Further when this preform was fiber drawn to a fiber of clad outside diameter 125 μm , the total length of the drawn fiber was 120 km; and the average transmission loss at wavelength 1.30 μm was 0.327 dB/km and the average transmission loss at 1.55 μm was 0.187 dB/km—a high quality fiber was obtained.

Implementation Example 2

Using the same size glass rod for core and the glass pipe for the clad prepared similarly to Implementation Example 1, an preform for optical fiber was manufactured by collapsing the assembly under the identical conditions in Implementation Example 1 with the exception that the heater length was set to be 100 mm. The obtained preform was measured by the preform analyzer similarly to Implementation Example 1: the fluctuation of the clad diameter/core diameter was verified to be $\pm 0.75\%$. For example, in the communication optical fiber for wavelength 1.30 μm , its cut-off wavelength has to be set to 1.10—1.29 μm . Here, the cut-off wavelength is inversely proportional to the clad diameter/core diameter. Therefore, for realizing the aforementioned cut-off wavelength of 1.10—1.29 μm , the fluctuation of the clad diameter/core diameter have to be suppressed to be below 8%. Accordingly, the heater length of 100 mm in this case is the limit of the range. It is clear that the heater length is preferably to be set to be less than 100 mm.

Comparison Example 1

Using the same size glass rod for core and the glass pipe for the clad prepared similarly to Implementation Example 1, an preform for optical fiber was manufactured by collapsing the assembly into one body by using a heater of 60 mm in length. The conditions here were heater temperature 1800°C; the (preform) ascending speed 20 mm/min; the atmospheric pressure difference by the pressure reduction was set to 300 mm

H₂O in carrying out the operation. The obtained preform was investigated by the preform analyzer. The fluctuation of the clad diameter/core diameter was less than $\pm 1\%$ --a good value. However, there were many blisters present in the interface between the core and the clad. The portion which can be used for fiber drawing was only 1/3 (or 1/2; not clearly copied) of the total length. This good portion was fiber drawn into a fiber of outside diameter 125 μm to obtain a total length of 35 km (comparison article).

The transmission losses at wavelength 1.30 μm and 1.55 μm of this fiber were 0.36 dB/km and 0.24 dB/km, respectively. This performance is inferior compared to the fiber of the present invention based on Implementation Example 1.

Furthermore, the comparison article and the fiber of the present invention of Implementation Example 1 were exposed to hydrogen (H₂) at 1 atmospheric pressure atmosphere at room temperature. There was no change in the transmission loss spectrum of the present invention fiber. However, in the comparison article fiber, the absorption peak by the hydroxyl group of wavelength 1.39 μm increased from 0.5 dB/km to 3.5 dB/km. And it became clear that even in the hydrogen durability performance, it was inferior.

From the aforementioned Implementation Example 1 and 2 and the Comparison Example 1, it is clear that for realizing a large type and high quality preform for optical fiber by the simple rod in tube manufacturing method, it is effective to use the method of collapsing the assembly to one body by using an electric furnace under the conditions specified in the present invention.

[Effect of the Invention]

As described above, in the present invention when the preform for optical fiber possessing the core and the clad by the rod in tube method, for the heating means, an electric furnace is used to achieve the production of a large type preform. Its practical application ability is great; it is economical and enhancement in process efficiency is achieved. Further, this is a method which is extremely advantageous industrially for manufacturing a high quality preform for optical fiber.

4. Brief Explanation of Figures

Fig 1 and Fig 2 are outline of illustration diagrams showing the implementation modes of the present invention. In Fig 1, an example is shown that the operation is carried out with the atmospheric gas being introduced. In Fig 2, an example is shown that the operation is carried out with the pressure reduced (hollow portion of the pipe) while the atmospheric gas is being introduced.

Fig 1

Atmospheric gas

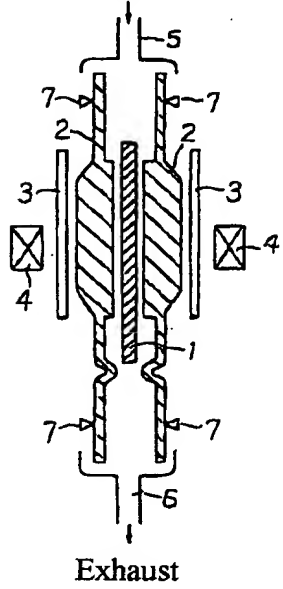
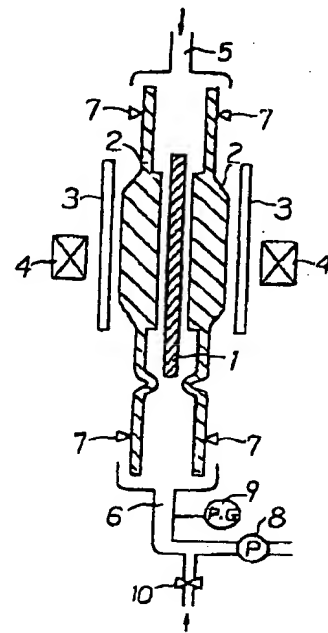


Fig 2

Cl₂ or chlorine compound gas, etc.

Gas for pressure adjustment

⑫ 公開特許公報(A)

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⑬発明の名称 光ファイバ用母材の製造方法

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明 細 書

1. 発明の名称

光ファイバ用母材の製造方法

2. 特許請求の範囲

- (1) 外径50mm以上のクラッド用ガラスパイプの中空部分に、該クラッド用ガラスパイプの内径より小さい直径のコア用ガラスロッドを挿入し、これを長さ30mm以上100mm以下のヒータを有する電気炉中でヒータ温度1800～2000℃で5mm/分以上20mm/分以下の速度でトウパースさせながら加熱することにより、該クラッド用ガラスパイプと該コア用ガラスロッドを一体化してコア及びクラッドを有してなるガラスロッドを得ることを特徴とする光ファイバ用母材の製造方法。
- (2) 加熱は該クラッド用ガラスパイプの中空部分内を大気圧差50～500mmHgの減圧に行なう特許請求の範囲第1項記載の光ファイバ用母材の製造方法。
- (3) 加熱は該クラッド用ガラスパイプの中空部

分内の雰囲気を変気又は塩素化合物ガスを含む雰囲気として行なう特許請求の範囲第1項又は第2項記載の光ファイバ用母材の製造方法。

- (4) 該コア用ガラスロッドが純石英からなり、かつ該クラッド用ガラスパイプが純石英に対する屈折率差が0.25%以上低い屈折率のフッ素添加石英ガラスからなるものである特許請求の範囲第1項記載の光ファイバ用母材の製造方法。

3. 発明の詳細な説明

〔産業上の利用分野〕

本発明は大型の光ファイバ用母材の製造方法に係わるものである。

〔従来の技術〕

光ファイバ用母材の製造方法において、クラッド材となる管の中にクラッド材よりも高屈折率のコア用ガラスロッドを挿入し、加熱し、中実化して光ファイバ用母材を製造するロッドインチューブ法は代表的な製造方法として知られ

ている。

しかし、この方法はコア材とクラッド材の界面に欠陥(気泡、不純物等)が残り易く、光ファイバとした時に、光損失が大きく現れるという欠点があつた。これを解決する方法として、特公昭59-6261、特公昭58-52935各号公報において、コア材とクラッド材との溶着・中実化前に、管とロッドとの間隙に電相処理剤を流し、コア材が変形しない温度500～1600℃の範囲にて加熱前処理する方法が提案されている。

〔発明が解決しようとする問題点〕

ところで、従来のロッドインチューブ法では、コア材とクラッド材を中実化する加熱手段としては、酸水素炎が用いられていた。しかし、酸水素炎による加熱はその効率に限度があり、せいぜい母材外径が30mm程度のものでしか得られなかつた。

近年、光ファイバ製造においても大量生産による低コスト化が推進されており、大型母材の

作製が検討されているが、上記のような母材外径における制限は一つのネックとなっており、早急な解決が望まれている。

これに対し酸水素炎に比してより加熱効率が高く、しかも母材への水酸基の混入の危険のない電気炉例えば抵抗炉等を用いて中実化することが考えられるが、抵抗炉を用いると母材の変形が著しく、未だこの方法の実用化はされていなかった。

本発明は以上のような現状に鑑みてなされたもので、ロッドインチューブ法における加熱手段として電気炉を用い、大型母材を変形なく製造することを可能とする実用性大で経済上、工程上の効率を向上した方法を提供しようとするものである。

〔問題点を解決するための手段及び作用〕

本発明者らはロッドインチューブ法によるコアとクラッドの加熱一体化手段として電気炉を用いても、母材変形をきたさずに製造できる条件について、鋭意研究し、コア材、クラッド材

それぞれのサイズ、さらに加熱の仕方、条件を特定の組合せにすることではじめて、前記の目的が達成できることを見出した。

すなわち、本発明は外径50mm以上のクラッド用ガラスパイプの中空部分に、該クラッド用ガラスパイプの内径より小さい直径のコア用ガラスロッドを挿入し、これを長さ30mm以上100mm以下のヒータを有する電気炉中でヒータ温度1800～2000℃で5mm/分以上20mm/分以下の速度でトラバースさせながら加熱することにより、該クラッド用ガラスパイプと該コア用ガラスロッドを一体化してコア及びクラッドを有するガラスロッドを得ることを特徴とする光ファイバ用母材の製造方法である。

本発明の特に好ましい実施態様としては、加熱を該クラッド用ガラスパイプの中空部分内を大気圧差50～300mmH₂Oの差圧に行なう上記方法及び加熱を該クラッド用ガラスパイプの中空部分内雰囲気を増熱又は塩素化合物ガスを含有するものとして行なう上記方法が挙げら

れる。また本発明においてコア用ガラスロッドが純石英からなり、かつクラッド用ガラスパイプが純石英に対する比屈折率差が0.25%以上低い屈折率のフッ素添加石英ガラスからなるものは、特に好ましいコアとロッドの組合せである。

以下に本発明を図面を参照して具体的に説明する。第1図は本発明の一実施態様を示す概略説明図であつて、同図において1はコア用ガラスロッド、2はクラッド用ガラスパイプ、3は炉心管、4は電気炉例えば抵抗炉等のヒータ、5は雰囲気ガス導入口、6は排気口、7は母材把持及びトラバース用のチャックであり、図のようにクラッド用ガラスパイプ2の中空部内にコア用ガラスロッド1を設置して、チャック7により把持しトラバースしながら電気炉のヒータ4により両者を加熱一体化するのである。このときに雰囲気ガス導入口5から適宜雰囲気ガスを導入して行なう。

本発明においては、クラッド用ガラスパイプ

としては、コア用ガラスロッドより屈折率が低いガラスからなり、外径50mm以上のものが特に好ましい。外径が50mm未満では、電気炉のヒータ等からの不純物揮散によりクラッド用ガラスパイプ外壁が汚染された場合に、該パイプ外壁の影響があらわれ易くなるからである。そして、コア用ガラスロッドは該クラッド用ガラスパイプより高い屈折率のガラスからなり、その直径(外径)が該クラッド用ガラスパイプの内径より小さいものを用いる。

このようにコア用ガラスロッドとクラッド用ガラスパイプの好ましい組合せとしては、例えばコア用ガラスロッドが純石英であり、クラッド用ガラスパイプがフッ素添加石英ガラスである組合せが挙げられる。この理由は、フッ素添加石英ガラスは純石英ガラスよりも屈折率が小さく、かつ光伝送損失の劣化を生じさせることが少ないため、石英ガラスコアに対して極めて優れたクラッド材とすることができるからである。また、この組合せによりシングルモードフ

ファイバを作製するには、フッ素添加石英ガラスの屈折率を純石英ガラスの屈折率よりも比屈折率差で0.25%以上低くすることが好ましい。これにより、曲げ損失に強く、伝送損失の安定したファイバとすることができる。なお、このようにコア用ガラスロッド、クラッド用ガラスパイプは、VAD法、OVD法、MCVD法、ゾルゲル法、プレス法その他の公知技術により製造される。

本発明において加熱源とする電気炉例えば電気抵抗炉等はそのヒータが長さ100mm以下30mm以上であることが好ましく、ヒータ温度は1800~2000℃に加熱しておき、5mm/分以上20mm/分以下の速度でトラベースさせる。ヒータ長さが100mmを越えると母材変形が起きるし、30mm未満ではクラッド用ガラスパイプ外壁の温度が高温となつても、十分にクラッド用ガラスパイプ内壁またはコア用ロッド表面の温度を上げにくくなるからである。ヒータ温度が1800℃未満では、同様にクラッド用ガ

ラスパイプ内壁表面温度、コア用ロッド表面温度が必要温度にあがらず好ましくなく、また、2000℃を越えるとクラッド用ガラスパイプの変形が急激に大きくなる。トラベース速度が5mm/分未満では母材に変形をきたし、20mm/分を越えると母材に気泡が発生して、この母材から得られるファイバの特性が劣化するため好ましくない。

本発明においては加熱の際にクラッド用ガラスパイプの中空部内を大気圧 $50 \sim 300$ mm H₂Oの減圧として行なうことが好ましい。特に好ましくは100~300mm H₂Oの減圧である。50mm H₂O未満では充分に中実化を行ない難く、300mm H₂Oを越えるとファイバの特性が劣化するからである。

また加熱の際にクラッド用ガラスパイプの中空部分内を塩素又は塩素化合物ガスを含む雰囲気として行なうことは特に好ましい。これによりコア用ガラスロッドとクラッド用ガラスパイプの脱水が充分に行われ、残留水酸基量が極度

に低減されたガラスロッドを得ることができる。このときの塩素又は塩素化合物ガスとしては、例えばCl₂、CCl₄、SOCl₂等を用いることができる。

図2図に塩素又は塩素化合物ガス雰囲気なかつ減圧にする際の装置構成を示すが第1図の構成に加えて、排気口部分6に減圧手段8を設ける。また排気口6と減圧手段8の間には圧力計9と圧調整用ガス導入用バルブ10を設けておく。雰囲気ガス導入口5よりクラッド用ガラスパイプ2の中空部内に塩素又は塩素化合物ガスを導入しつつ、減圧手段8により減圧するが、バルブ10より圧調整用ガスの流量をコントロールして、圧力計9の圧を設定圧とする。このように圧調整用ガスとしては、例えばN₂、Ar、He等の不活性ガス、塩素又はSOCl₂、CCl₄等の塩素化合物ガスが用いられる。

以上のように行なうことにより、本発明は従来よりも太径で高品質の光ファイバ用母材を、電気炉を用いたロッドインチューブ法により変

形等なく製造できるが、以下に実施例、比較例を挙げて具体的手段と本発明の効果を示す。

〔実施例〕

実施例 1

コア用ガラスロッドとしては、VAD法（気相軸付法）により作製した純石英ガラスロッド（ $5\text{mm}\phi \times 500\text{mm}$ ）を用い、クラッド用ガラスパイプとしてはフッ素（F）が11重量%添加された石英ガラスパイプ（外径 $75\text{mm}\phi$ 、内径 $10\text{mm}\phi$ 、長さ 400mm ）を用いて、第2図の構成で本発明により加熱一体化を行なった。このとき、長さ 60mm のヒータを用い、ヒータ温度 1970°C の状態でクラッド用パイプの孔内に雰囲気ガスとして $1\text{L}/\text{分}$ の Cl_2 を流し、かつクラッド用ガラスパイプの孔内を大気圧差 250mmHg に減圧しながら、母材を $8\text{mm}/\text{分}$ の速度で上昇させ加熱一体化した。上昇終了後、得られた母材を炉から取り出し、延伸した後、プリフォーム・アナライザを用いてコア径及びクラッド径の測定を行なった。長手方向

に偏かに変動はあつたものの、クラッド径／コア径の変動は1%以下であり、コア・クラッド界面に気泡の無い、良好な母材が得られた。コア・クラッドの屈折率差は 0.50% であつた。

なお、この母材をクラッド径 $125\mu\text{m}$ のファイバに線引したところ、線引長は全長 120km であり、波長 $130\mu\text{m}$ における伝送ロスに平均 $0.327\text{dB}/\text{km}$ 、 $1.55\mu\text{m}$ の伝送ロスは平均 $0.187\text{dB}/\text{km}$ という高品質なファイバであつた。

実施例 2

実施例1と同じに作製した同サイズのコア用ガラスロッドとクラッド用ガラスパイプを用いて、ヒータ長を 100mm とした以外は、実施例1と同じ条件でコアコアスして光ファイバ用母材を製造した。得られた母材を実施例1と同様にプリフォーム・アナライザで測定したところ、クラッド径／コア径には $\pm 7.5\%$ の変動が認められた。例えば、波長 $130\mu\text{m}$ 用の通信用光ファイバでは、そのカットオフ波長を 110

$\sim 1.29\mu\text{m}$ にする必要がある。ここで、カットオフ波長は母材のクラッド径／コア径に反比例するため、上記の $110\sim 1.29\mu\text{m}$ というカットオフ波長を実現するためにはクラッド径／コア径の変動を $\pm 8\%$ 以下に抑える必要がある。従つて、ヒータの長さが 100mm の場合ではこの範囲に於いての限界であり、ヒータ長を 100mm 以下とすることが好ましいことがわかる。

比較例 1

実施例1と同じに作製した同サイズのコア用ガラスロッドとクラッド用ガラスパイプを用いて、長さ 60mm のヒータを用いて加熱一体化を行つたが、このときの条件を、ヒータ温度 1800°C 、上昇速度 $20\text{mm}/\text{分}$ 、減圧による大気圧との差を 300mmHg として行つた。得られた母材（比較品）について実施例1と同様に、プリフォーム・アナライザを用いてクラッド径／コア径を調べたところ、その変動は $\pm 1\%$ 未満と小さく良好であつた。しかしながら、コ

ア・クラッド界面に微小な気泡が多く、線引用プリフォームとして使用できる部分は、全体の 5% 程度であつた。この良好な部分を外径 $125\mu\text{m}$ のファイバに線引して全長 35km のファイバ（比較品）を得た。

該ファイバの波長 $130\mu\text{m}$ 、 $1.55\mu\text{m}$ における伝送ロスはそれぞれ $0.56\text{dB}/\text{km}$ 、 $0.24\text{dB}/\text{km}$ であり、実施例1による本発明ファイバと比較すると、その特性は劣下していた。

さらに、この比較品ファイバと実施例1の本発明ファイバを、室温下、水素（ H_2 ）1気圧雰囲気中に1週間曝したところ、本発明ファイバの伝送ロスのスペクトラムには何の変化も無かつたが、比較品ファイバでは波長 $139\mu\text{m}$ の水酸基による吸収ピークが $0.5\text{dB}/\text{km}$ から $3.5\text{dB}/\text{km}$ に増加しており、耐水素特性に於ても劣つていたことがわかつた。

上記実施例1及び2と比較例1の結果から、大型で高品質な光ファイバ用母材を簡単なロットインテグレーション法で製造することを實現するに

